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ANNUAL TECHNICAL REPORT 1 JUNE 1980-31 MAY 1981.(U)

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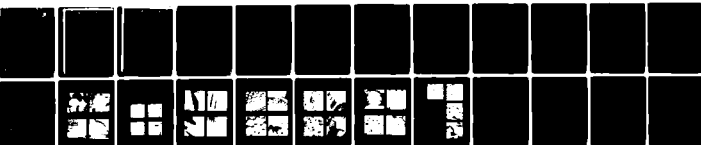
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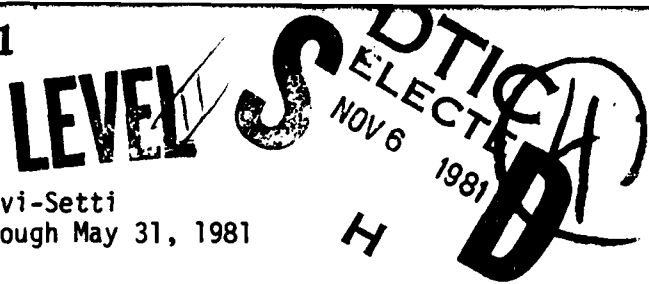
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ANNUAL TECHNICAL REPORT

AFOSR Contract No. F49620-80-C-0074
Principal Investigator: Riccardo Levi-Setti
Contract Period: June 01, 1980 through May 31, 1981

Research Objectives for the First Year

The University of Chicago (UC) and Hughes Research Laboratories (HRL) collaborative program aims at the development and construction of two 60 keV high resolution (10-100 Å), high current density (1A/cm²) ion microscopes/microprobes over a three year period. A pictorial summary of the organization of the project and its funding is contained in Fig. 1. Two sets of coordinated technical and scientific tasks form an integral part of the UC-HRL program. A phasing diagram for these tasks is given in Fig. 2, also showing the repartitioning of DOD (through AFOSR) and NSF support among the various tasks.

The research objectives for the first year of the program, comprehensive of both the UC and HRL effort, are incorporated in the following main tasks:

1. Design studies using the existing ion probes.

At UC these comprised the installation of an HRL-built EHD liquid gallium source in the existing Chicago STIM. The determination of the energy spectrum and beam composition of the source, and the assessment of the performance of counting detector electronics under high counting rates.

At HRL this task consisted in further exploration of the factors limiting the resolution which can be achieved with one of their existing single-lens focusing columns.

The results of these parallel investigations were to help in finalizing several aspects of the design and instrumentation relative to the new high performance ion probes to be constructed.

2. New ion microscope development.

The initial phase of this task, common to both UC and HRL, involved the performance of optical calculations leading to an optimized microscope design, the preparation of a preliminary instrument design and of part of the engineering design, and the choice and procurements of components and instrumentation.

Close contact between the UC and HRL team was to be maintained through periodic meetings during this critical decision-making stage.

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) The University of Chicago (UC) and Hughes Research Laboratories (HRL) collaborative program aims at the development and construction of two 60 keV high resolution, high current density ion microscopes/microprobes over a three year period. Using existing low resolution probes at UC and HRL, design studies have been conducted to determine: (a) the energy spectra of gallium ions emitted by a liquid metal (LM) source; (b) the optimum operating condition of the source; (c) the limitations of various			

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image display methods at high and low probe currents; (d) the current yields of several metal ions from LM sources. Optical calculations have been performed to optimize the design of the new microprobe column. An overall system design has been formulated and assembly and engineering drawings completed. Equipment and parts to be procured have been selected. The UC existing probe has been used to continue basic research studies of ion-solid interactions. These include the interaction of molecular hydrogen ions with thin solid foils, ion-induced secondary electron emission, and damage to light element targets under gallium ion bombardment.

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3. Study of physical processes yielding image contrast in ion microscopy.

This UC task was designed to pursue an active program of basic investigations of the ion solid interaction, using the existing UC-STIM, in parallel with the new ion microscope development. It was deemed essential to bring forward the study of topics suitable for the proper training of graduate students aiming at their Ph.D. degree in Physics, in areas seldom represented in an academic atmosphere. Basic to problems in microstructure physics or microscience at large, these included the study of the transmission of hydrogen and gallium ions through solid targets, secondary electrons and ion emission under gallium ion bombardment, and of target damage during ion imaging.

Status of the Research Effort

Significant progress has been made toward completion of the tasks outlined above for the first year of our program. The following paragraphs give summary descriptions of the main accomplishments within the scope of each task.

1. Progress on design studies using the existing ion probes.

- a) After constructing an appropriate tantalum housing, an HRL-built EHD liquid gallium source has been installed in the UC-STIM. The new gallium probe has been operational since January 1981 and has already provided valuable insights on many issues of concern. We have studied the energy spectra, and beam composition of the source. Fig. 3 shows two momentum spectra obtained with the STIM magnetic sector spectrometer. The energy spread of the emitted Ga^+ ions is characterized by a full width at half maximum in the range 7 - 50 eV for source currents in the range 0.6 - 50 μA as shown in Fig. 4a. This information sets on a firm basis the design specifications of the new ion probes in regard to the effect of chromatic aberrations. It clearly indicates (Fig. 4b) the existence of an optimum operating condition which maximizes beam current while minimizing chromatic aberration.
- b) We have explored the limitations in the pulse-mode image display at high counting rates. This in order to simulate the conditions which will prevail at the highest resolution achievable with the new ion probe. We have determined that good quality micrographs with linear response can be obtained at counting rates up to ~ 2 MHz, while saturation and loss of contrast sets in at higher rates. Figs. 5, 6, and 7 show images of a variety of structures, obtained by secondary electrons

with a channel multiplier detector, under 60 keV Ga^+ ion bombardment.

We have also explored the analog-mode of image display, after constructing a display system which uses the current signal from the channel multiplier. High-quality images reaching the theoretical resolution limit of the one-lens column ($\sim 1000\text{\AA}$) for a particular optical aperture setting, have been obtained by this approach. This can yield fast interlaced scans (Figs 8 and 9) or single slow scans for maximum resolution (Fig. 10). The exposure times for the micrographs shown are typically 8 - 10 seconds.

- c) At HRL the development of new LM ion sources compatible with the requirements of the new high resolution ion microprobes has been extended to demonstrate the excellent performance of an Au-Si eutectic, yielding an intense silicon ion beam at 400°C . Excellent quality micrographs obtained in the HRL probe with this source are shown in Fig. 11. The specimen, supplied by UC, is similar to those illustrated in Fig. 5 and 8, and serves the purpose of comparing the performance of the HRL and UC instruments.
- d) A new test column, similar to the existing ones, is being built at HRL as part of the industry participation in the NSF-supported portion of the program. A fully dedicated instrument is in fact needed to carry out the planned investigation by HRL of the focusing limits of one-lens systems.

We plan to report on the results outlined above in a joint UC-HRL publication (see Publications (b) 8) and two papers to be presented at a Symposium (see Interactions (a) 6,7).

2. Progress on New Ion Microscope Development.

- a) Optical calculations. The original preliminary optimization of the column design for the new microprobes has been considerably extended, much along the lines set forth in the correspondence between the Principal Investigator and Dr. J.H.Harris and B.A.Wilcox of NSF, in answer to several comments and suggestions on the part of the NSF referees. Particular attention has been given to explore optimal configurations for longer working distances and larger demagnifications than previously

considered. By and large, the new exploration confirms the soundness of the basic design originally proposed. In more detail, the performance of the ion microprobe focusing column was analyzed to determine the expected focused spot size as a function of the relevant operating parameters (ion energy spread ΔV , source extraction voltage V_{EXT} , and working distance d_{WORK}). The results show that the calculated spot size d_{SPOT} is between 10 Å and 100 Å for anticipated values of the operating parameters. The column is designed so as to:

- o Decouple the source extraction voltage from the final probe voltage
- o Maintain a fixed object and image position while varying source and probe voltages over an extended range
- o Minimize the chromatic aberration of the column
- o Provide a crossover where a small aperture could be placed without affecting the transmission of the column. Such an aperture would stop a large fraction of the residue of nonfocused, neutralized and energy degraded beam particles that would still originate in the source region, in particular when working with a GFI source operating at several mTorr.

Figure 12 shows a schematic drawing of the two-lens electrostatic focusing column. The first lens (nearest the tip) is an asymmetric lens which first decelerates and then accelerates the beam to the final energy. This lens images the emitting tip to form an intermediate crossover. The final lens serves to demagnify the intermediate crossover formed by the first lens to provide the focused beam spot. Lens aberrations and column performance were calculated for a tip-lens distance of 1.6 cm and a total column length of ~21 cm. For all these calculations the intermediate crossover was maintained 2 cm downstream of the first lens by suitably adjusting the intermediate electrode potential V_1 . It was found that the initially decelerating mode ($V_1 < V_{EXT}$) for the first lens provided the best overall performance.

Since the focusing column is designed to operate with the gas-field-ionization (GFI) and liquid-metal (LM) ion sources, our analyses included both cases. The GFI source produces non-damaging H_2^+ ions which have been found very useful for transmission microscopy of thin specimens. In the transmission mode, the detector apparatus is located beneath the specimen and short working distances (e.g., $d_{WORK} = 1.2$ cm) are feasible. This

source has a relatively low energy spread (~ 2 eV). The extraction voltage can vary from 5 to 30 kV depending on the radius of the field-emitting tip.

Figure 13 shows the calculated spot size for the GFI source. The spot size is limited by diffraction at small acceptance angles ($\alpha_1 < 5 \times 10^{-4}$ rad) and by chromatic aberration for large angles. Minimum spot size occurs at $\alpha_1 \sim 8 \times 10^{-5}$ rad which corresponds to an expected current of 2×10^{-14} A; spot size is not sensitive to V_{EXT} provided the first lens is adjusted appropriately. Minimum spot size is attained for the smallest working distance (1.2 cm) as expected. However, d_{SPOT} only increases by $\sim 2\times$ when the working distance is increased to 5.6 cm. This latter condition is appropriate for reflection spectrometry techniques which require placing detectors between the final lens and the specimen.

LM sources typically emit heavy ions which sputter a specimen yielding characteristic secondary ions and electrons. The extraction voltage range is 5 to 10 kV, and an energy spread of 10 eV is common. Figure 14 shows the calculated spot size for an LM gallium source. The virtual source size is not well known for LM sources and was assumed zero in these calculations. The diffraction limit is at $\sim 10^{-5}$ rad for the heavier ions, and again chromatic aberration is limiting at larger values of α_1 . Minimum spot sizes occur at $\sim 1.5 \times 10^{-5}$ rad, where the expected spot current would be $\sim 2 \times 10^{-14}$ A. We anticipate using the larger working distance (5.6 cm) for SIMS with LM sources.

- b) New ion microprobe design. Figure 15 presents the design for the ion microprobe that has evolved after extensive deliberations by both HRL and UC investigators. The principal factors which have dictated this design are:
- o Ease and safety of operation and maintenance
 - o Ability to meet performance goals
 - o Component selection for reliability
 - o Type and size of vacuum pumps
 - o Interface requirements to the GFI/LM sources and UC-STIM spectrometer.

The mechanical system is small with approximate overall dimensions of 72"L x 40"W x 66"H. The main support table provides rigid mechanical integration of the system components and a low center of gravity for increased stability. Vibration isolators are used to decouple the table

from the laboratory floor and also provide a means of leveling the system. A combination of ion and sorption vacuum pumps eliminates the possibility of pump-caused vibration and backstreaming of oil. High-conductance transition pieces connect the pumping system to the ion probe column. The system pumpdown sequence is manually controlled with hand-operated valves.

The ion microprobe column and UC-STIM spectrometer occupy the center of the main support table. The STIM spectrometer magnet is supported by the table and its input port mates with the interface plate at the base of the column.

Power supplies to operate the system have been identified. A rack layout has been made which when integrated with the system's mechanical drawing, completes the overall system design.

Figure 16 is a more detailed view of the ion microprobe column which shows the major mechanical details. The column vacuum envelope will be manufactured from non-magnetic 304 stainless steel. The vacuum chamber design follows accepted practice of internal welds, electropolishing of interior surfaces and elimination of organic seals to insure low ultimate pressures are achieved in a reasonable time. System components are mated to the column using ConFlat ^(R) flanges. Moveable seals such as X-Y source positioning are provided by flexible bellows or Viton o-rings, so that the system can be baked out to 150°C.

The high voltage feedthroughs for the system are field proven commercial feedthroughs and are conservative in design. Internally, the high voltage isolation between lens elements is provided by high purity alumina ceramic. These insulators will be custom manufactured and conservatively stressed. The GFI or LM sources interface to the column via a ConFlat flange supported on a thrust bearing. This flange allows the source to align with the optics by means of micrometer-type X-Y adjustment heads. The column's optics and deflection elements are integrated into a module which can be removed from the column for pre-alignment, greatly facilitating maintenance. The lens module fastens to a ring that is welded inside the column vacuum chamber. When the lens module is in place, the column is divided into two subchambers. These subchambers are separately pumped, thus, providing a differentially pumped column.

The UC-STIM spectrometer is bolted to a large ConFlat interface plate at the bottom of the target chamber. This plate can easily be changed or adapted to other diagnostic instrumentation.

- c) UC-HRL contacts. The two collaborating teams have maintained close contact throughout. Two UC investigators (RLS and TRF) have visited HRL in August 1980 and again in April 1981. Two HRL investigators (RLS and RPV) were at UC in December 1980.

3. Progress in the study of physical processes yielding image contrast in ion microscopy.

This part of the UC program has been maintained at a high level of productivity.

- a) Several studies, previously undertaken, of the transmission of molecular H_2^+ ions through thin solid foils, have now been completed. Part of the results have been published (Publications (a) 1, 2, 3, 4) or reported to conferences (Interactions (a) 1, 2, 3, 4, 5); several others are in the course of preparation (Publications (b) 5, 6, 7). A Ph.D. Thesis project by T. R. Fox has been completed.
- b) We have initiated studies of secondary electron emission under Ga^+ ion bombardment. The micrographs already shown in Figs. 5 - 10 are part of this study and indicate substantial differences in the contrast mechanisms effective under ion versus electron bombardment, in particular concerning the behaviour of insulators. A survey of the response of diverse chemical microstructures is in progress.
- c) Studies of damage induced by Ga^+ ion bombardment are also in progress. Fig. 17 illustrates quantitatively a particular example of damage, caused to a thin carbon foil and plastic supporting microgrid. In general, damage information is obtained as a byproduct of imaging and focusing tests and will accumulate at a substantial rate for a variety of target materials.

In conclusion, we feel that our progress since the inception of the present program has proceeded close to schedule and are confident in a very positive outcome of our overall effort. We wish to add that our experience in this initial phase of the collaboration between the UC and HRL teams has been most rewarding to both parties involved. A real spirit of reciprocally helpful competition has become established between the two groups, making for an exciting and productive research atmosphere.

Cumulative Chronological List of Written Publications in the Technical Journals

(a) Published and in press

1. H_2^+ Traversing Ultra-Thin Carbon Foils: Measurement of Recombination Yields at 12.5 and 25 keV/amu.
T. R. Fox; Nuclear Instruments and Methods 179 (1981) 407-10.
2. Molecular States from 25 keV/amu H_2^+ Traversing Ultra-Thin Carbon Foils.
R. Levi-Setti, T. R. Fox and K. Lam; Annals of the Israel Physical Society, in press.
3. H_2^+ Traversing Ultra-Thin Carbon Foils: Exiting Molecular States at 12.5 and 25 keV/amu.
T. R. Fox, K. Lam and R. Levi-Setti; to be published in Nuclear Instruments and Methods.
4. H_2^+ Traversing Ultra-Thin Carbon Foils: Cluster Effects in the Energy Loss at 12.5 and 25 keV/amu.
R. Levi-Setti, K. Lam and T. R. Fox; to be published in Nuclear Instruments and Methods.

(b) In course of preparation

5. H_2^+ Traversing Ultra-Thin Carbon Foils: The Energy Loss of Two Correlated Protons Below the Fermi Velocity.
K. Lam; to be published in Nuclear Instruments and Methods.
6. Internuclear Separation of Diproton Clusters Traversing Thin Carbon Foils Below the Fermi Velocity.
K. Lam; to be published in Nuclear Instruments and Methods.
7. Scanning Microscopy with Hydrogen Ions from a Field Ion Source.
T. R. Fox and R. Levi-Setti; to be published in the Journal of Vacuum Science and Technology.
8. Scanning Microscopy with Gallium and Silicon Ions from Electrohydrodynamic Liquid Metal Sources.
UC-HRL Collaboration; to be published in the Journal of Vacuum Science and Technology.

(c) Theses

1. Hydrogen Ion-Solid Interactions Near the Bohr Velocity.
Timothy R. Fox. Ph.D Thesis. The University of Chicago, December 1980.

Interactions (Coupling Activities)

(a) Spoken papers presented at Meetings, Conferences, Seminars.

1. Progress in the Development of a Field Ionization Scanning Transmission Ion Microscope.

R. Levi-Setti. T. R. Fox and K. Lam.

Paper presented at the Thirty-eighth Annual EMSA Meeting, Reno, Nevada, August 1980.

Abstract in Proc. 38th Ann. Meet, EMSA, pp 66-7. Ed. G. W. Bailey, Claitor's Publ. Div., Baton Rouge, 1980.

2. Information and Dose in the Scanning Transmission Ion Microscope

T. R. Fox and R. Levi-Setti.

Paper presented at the Thirty-eighth Annual EMSA Meeting, Reno, Nevada, August 1980.

Abstract in Proc. 38th Ann. Meet. EMSA, pp 232-3. Ed. G. W. Bailey, Claitor's Publ. Div., Baton Rouge, 1980.

3. Same Title as Publications (a) 2. Paper presented by R. Levi-Setti at the Bat-Sheva Seminar on Molecular Ions, Molecular Structure and Interaction with Matter. Israel, January 1981.

R. Levi-Setti also chaired two sessions at the Seminar.

4, 5. Same as Publications (b) 3, (b) 4. Papers presented by R. Levi-Setti at the Ninth International Conference on Atomic Collisions in Solids, Lyons, France, July 1981.

6. Scanning Microscopy with Gallium Ions from a Liquid Metal Source.

K. Lam, T. R. Fox and R. Levi-Setti. Paper to be presented at the Twenty-Eighth International Field Emission Symposium, The Oregon Graduate Center, July 1981.

Abstract to be published in Proceedings of Symposium.

7. Magnetic Prism Analysis of Practical Gallium Ion Probe from a Liquid Metal Source.

T. R. Fox, R. Levi-Setti, and K. Lam. Paper to be presented at the Twenty-eighth International Field Emission Symposium, the Oregon Graduate Center, July 1981.

HIGH RESOLUTION ION MICROPROBE PROGRAM

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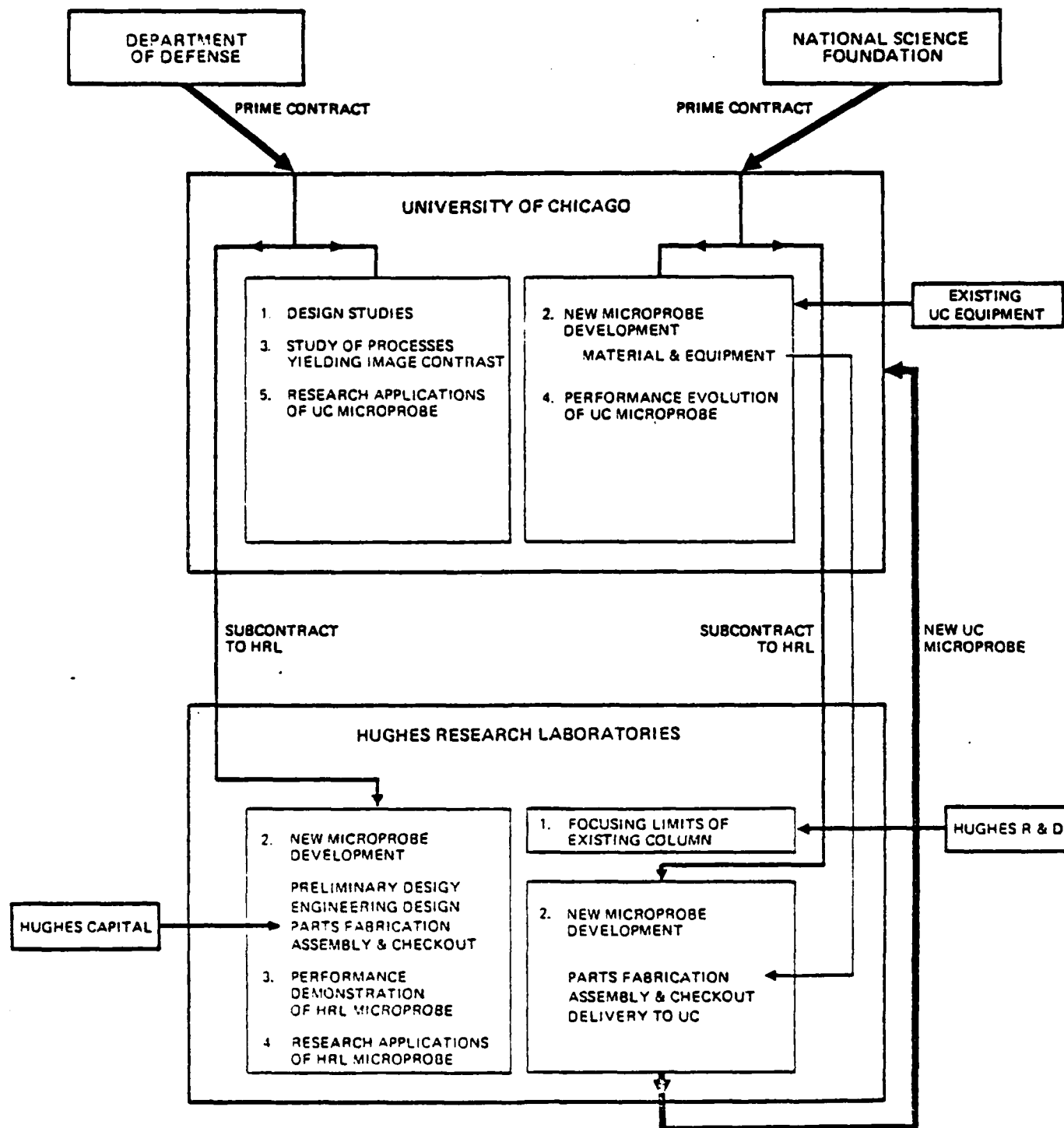


Fig. 1

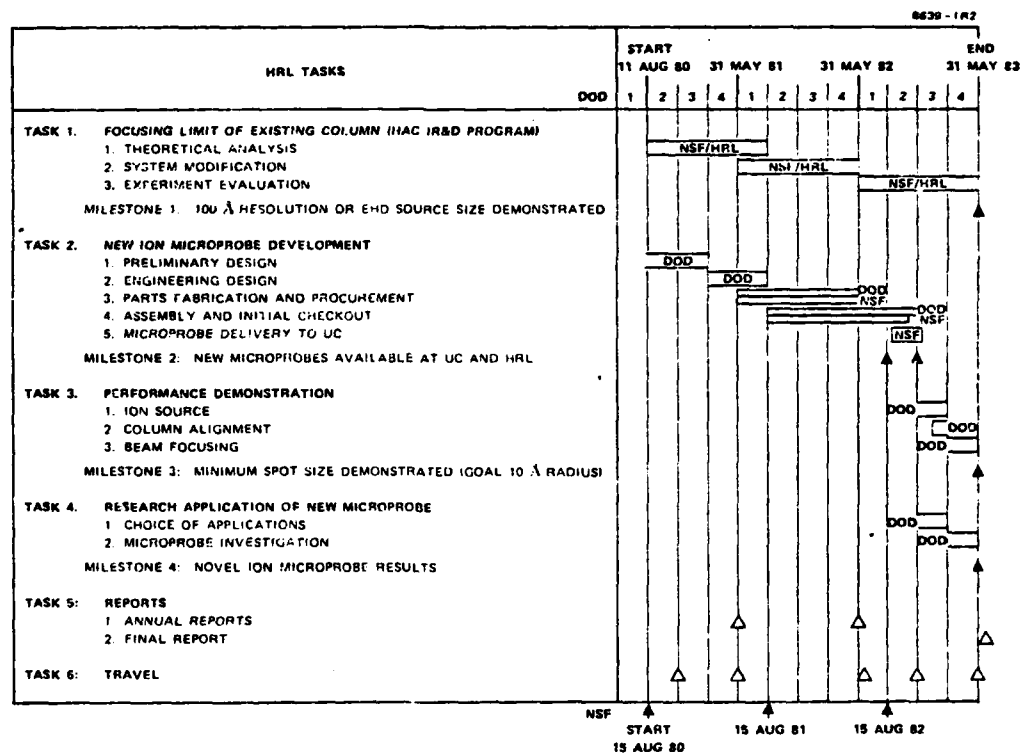
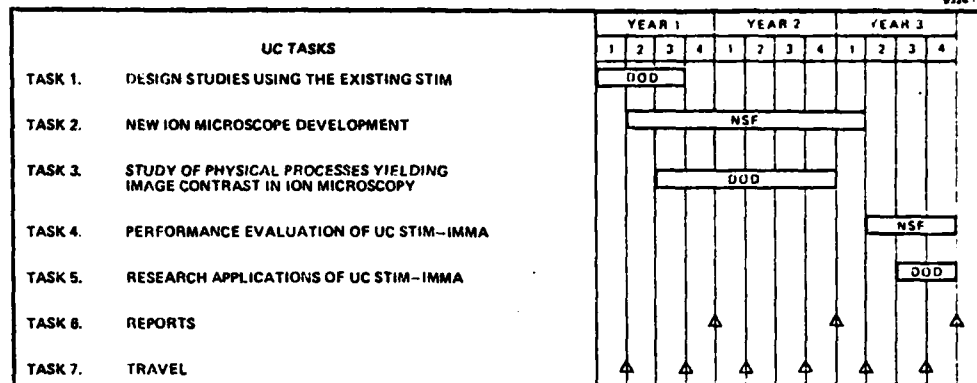
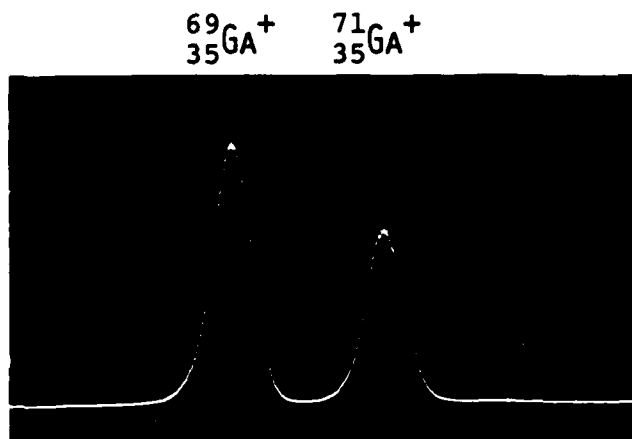


Fig. 2

GA⁺ MOMENTUM SPECTRA

$V_0 = 800 \text{ V}$
 $I_t = 2.1 \text{ } \mu\text{A}$
 $\text{FWHM} = 6.6 \text{ eV}$



$V_0 = 2000 \text{ V}$
 $I_t = 11 \text{ } \mu\text{A}$
 $\text{FWHM} = 21.4 \text{ eV}$

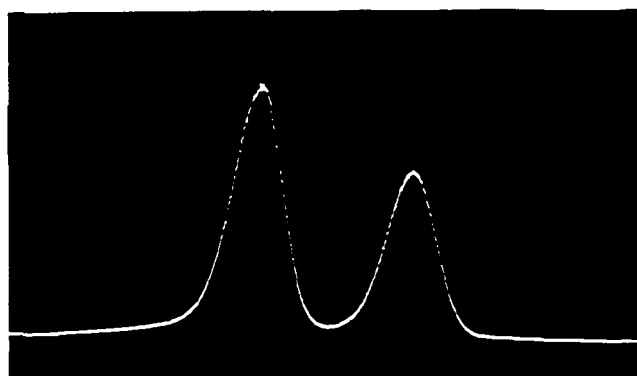


Fig. 3

Momentum analysis of gallium ion beam from an EHD liquid metal source installed in the UC-STIM. The gallium ions, extracted at 7 keV, are decelerated to 800 or 2000 eV respectively. The energy resolution is better than 1 eV. The ion energy spread is observed to increase with the source current.

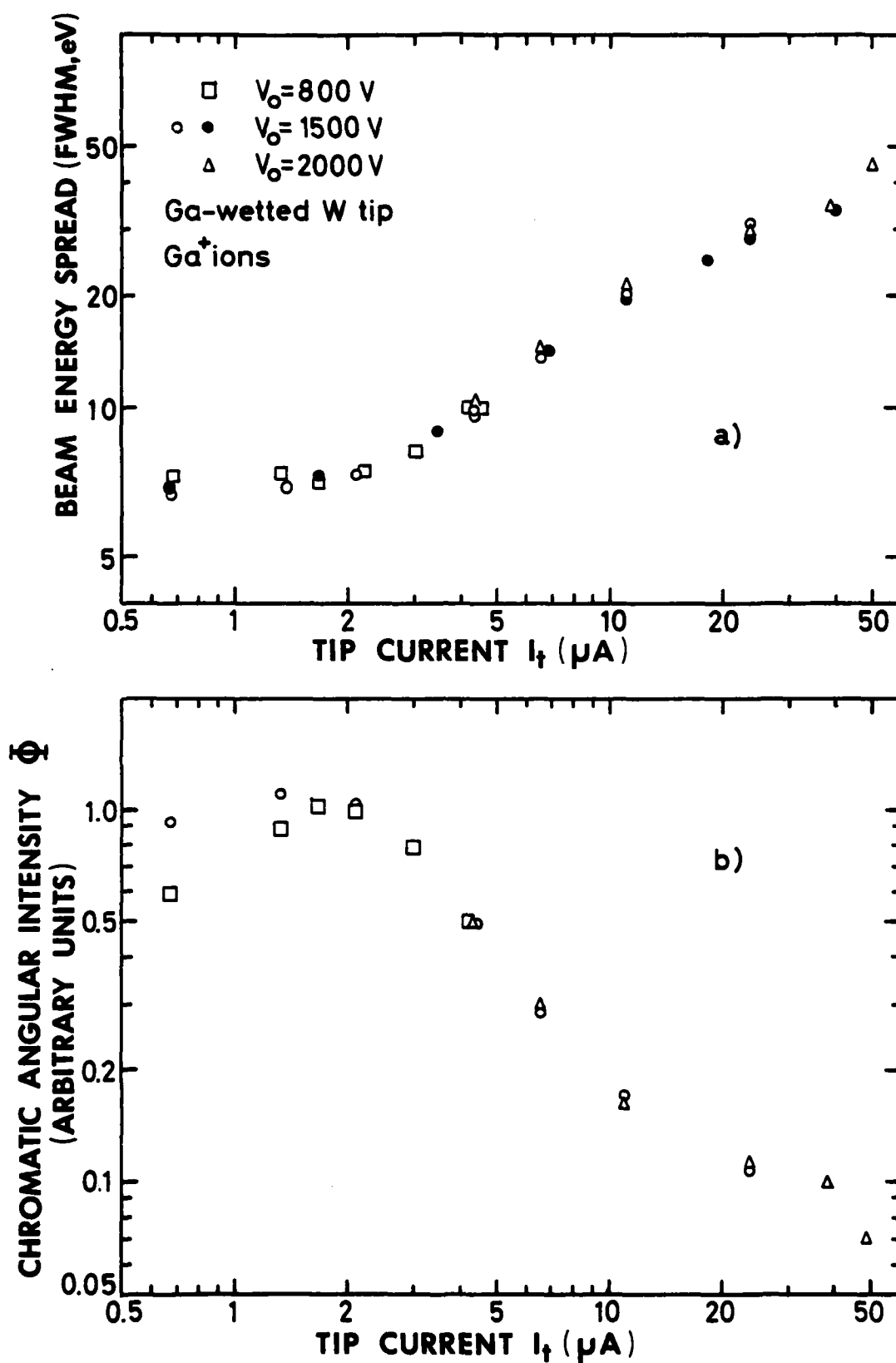
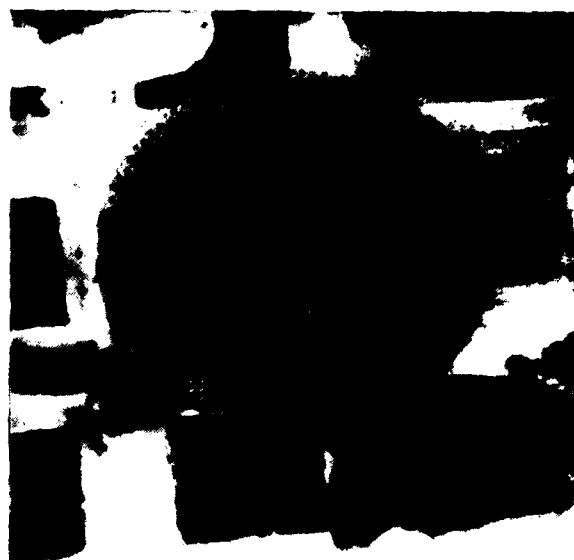


Fig. 4 a) Measured energy spreads of Ga^+ ions vs. tip current.
 b) Normalized chromatic angular intensity Φ vs. tip current. In a chromatic aberration-limited system, the probe current is proportional to Φ .

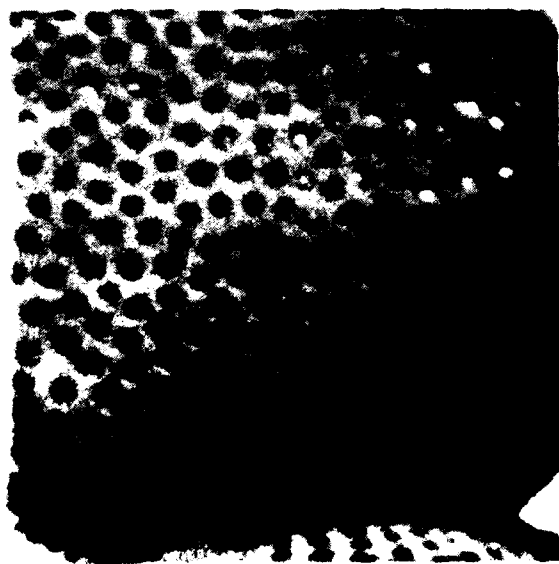
Ga^+ FOCUSED-ION-BEAM IMAGES
UNCOATED FOSSIL DIATOMS,
UC-SIM, PULSE-MODE DISPLAY, FAST SCAN



168 μm FULL SCALE



168 μm F.S.



67 μm F.S.



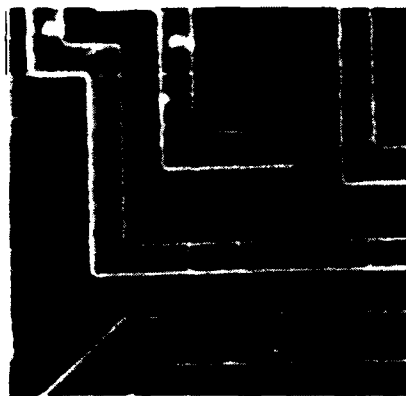
67 μm F.S.

Fig. 5 Micrographs of uncoated radiolarians (SiO_2) obtained with secondary electrons from a 60 keV Gallium ion beam in the UC-STIM. Ion source: HRL-built Ga/EHD liquid metal source. Source current: $2\mu\text{A}$. Probe current: $\sim 10^{-11}\text{A}$. Display: Counting mode, 512 lines, $\sim 4 \times 10^7$ counts, 10-20 sec/frame.

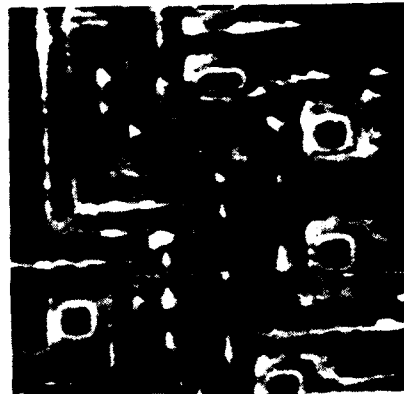
Ga^+ FOCUSED-ION-BEAM IMAGES

INTEGRATED CIRCUITS

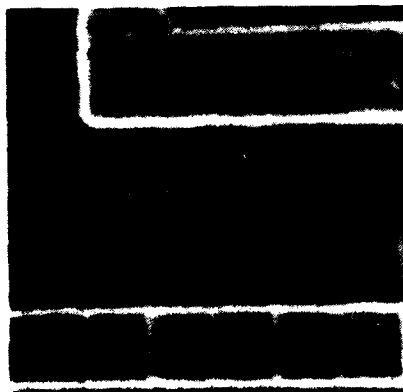
UC SIM. PULSE-MODE DISPLAY. FAST SCAN.



160 μm FULL SCALE.



160 μm F.S.



64 μm F.S.



64 μm F.S.

Fig. 6 Imaging of semiconductor devices by secondary electron emission with a 60 keV Ga^+ ion beam in the UC-STIM. The integrated circuits shown are covered by an insulating passivation layer.

Ga^+ FOCUSED-ION-BEAM IMAGES

FRACTURE IN Si

UC-SIM. PULSE-MODE DISPLAY. FAST SCAN



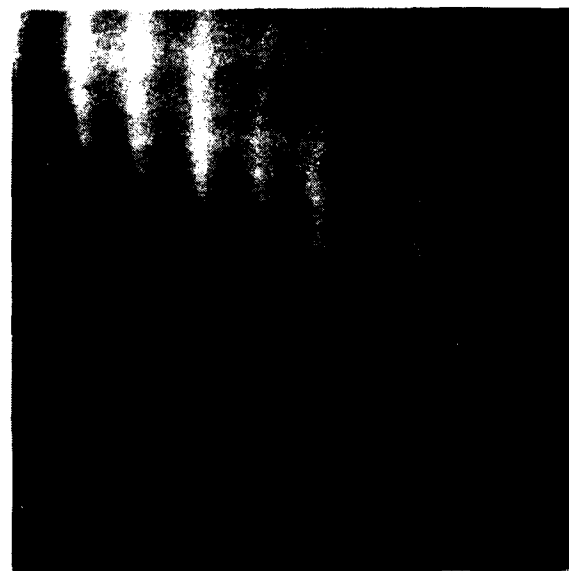
160 μm FULL SCALE



32 μm F.S.



32 μm F.S.



16 μm F.S.

Fig. 7

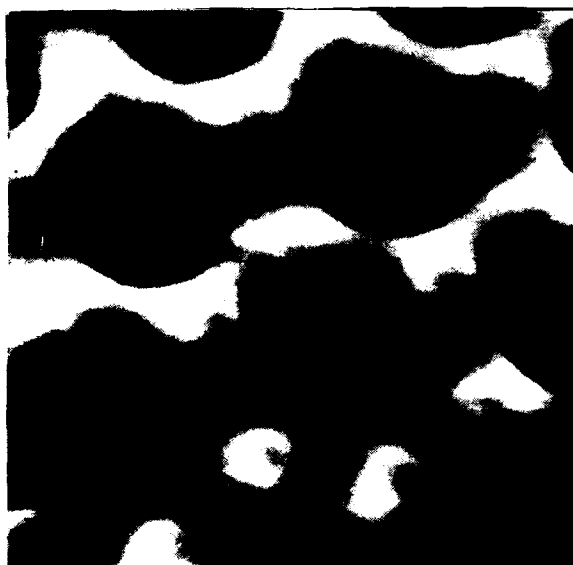
GA⁺ FOCUSED-ION-BEAM IMAGES
Ag-COATED FOSSIL DIATOMS
UC-SIM. ANALOG DISPLAY, FAST SCAN.



32 μm FULL SCALE.



32 μm F.S.



16 μm F.S.

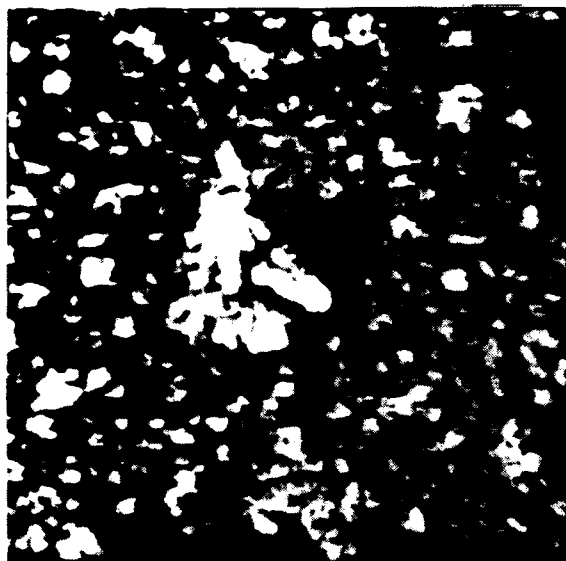


16 μm F.S.

GA⁺ FOCUSED-ION-BEAM IMAGES

UC-SIM. ANALOG DISPLAY. FAST SCAN

Ag CRYSTALS



156 μm FULL SCALE

FOSSIL DIATOM



68 μm F.S.



62 μm F.S.



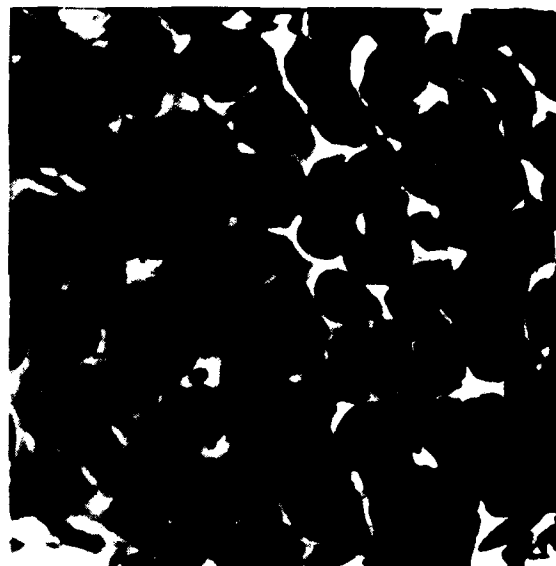
34 μm F.S.

Fig. 9

GA⁺ FOCUSED-ION-BEAM IMAGES
Ag-COATED FOSSIL RADIOLARIAN
UC-SIM. ANALOG DISPLAY. SLOW SCAN



108 μm FULL SCALE



70 μm F.S.

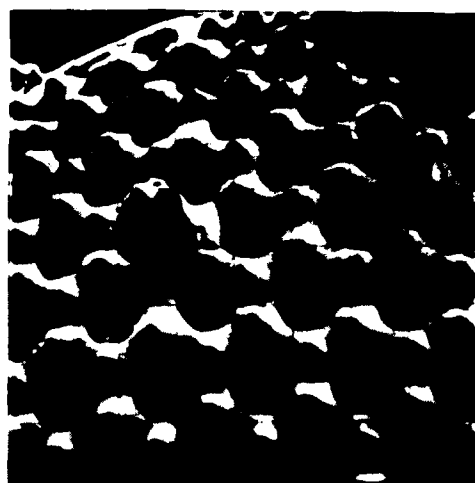


35 μm F.S.



17.5 μm F.S.

Fig. 10



5μm



2.5μm

Micrographs of a radiolarians
obtained with secondary electrons
from an 85 keV silicon ion beam
in the HRL-SIM.

Ion source: Au-Si eutectic, oper-
ated at 400° C.

Source current: 8 μA.

Probe current: $\approx 10^{-10}$ A.

Display: Analog mode, 1000 lines,
32 sec/frame.

Object material: Ag-coated SiO₂.

Note logo HRL imprinted on object
with ion beam, visible in the two
upper right micrographs.



0.5μm

Fig. 11

Fig. 12 Schematic Drawing of High Resolution Focusing Column

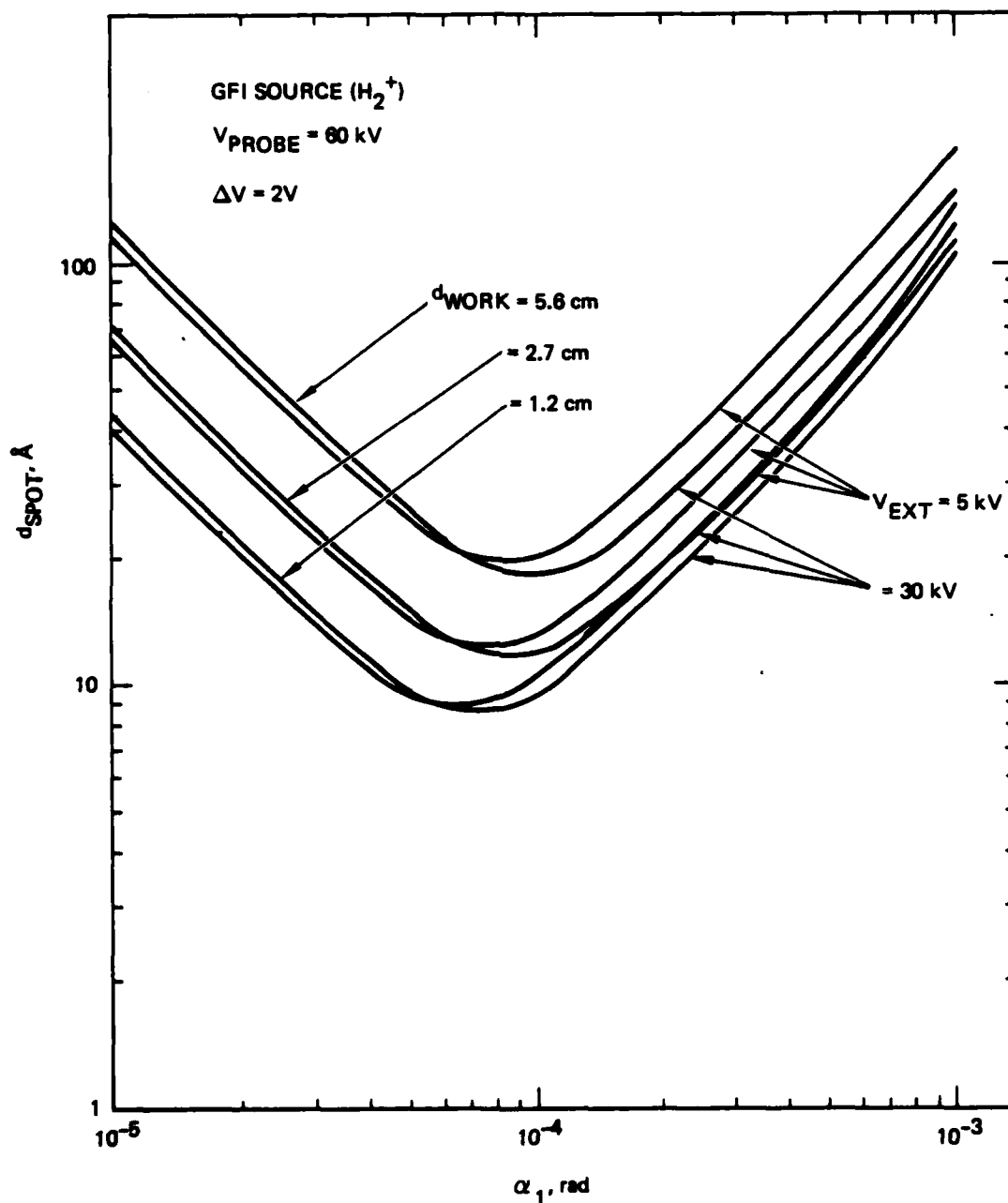


Fig. 13 Spot size as a function of aperture half-angle for GFI source omitting effect of virtual source size. At $V_{\text{EXT}} = 5 \text{ kV}$, the overall magnification of the system ranges between ~ 0.1 and ~ 0.4 , increasing with d_{WORK} . At $V_{\text{EXT}} = 30 \text{ kV}$, such range becomes $\sim 0.3 - 0.7$.

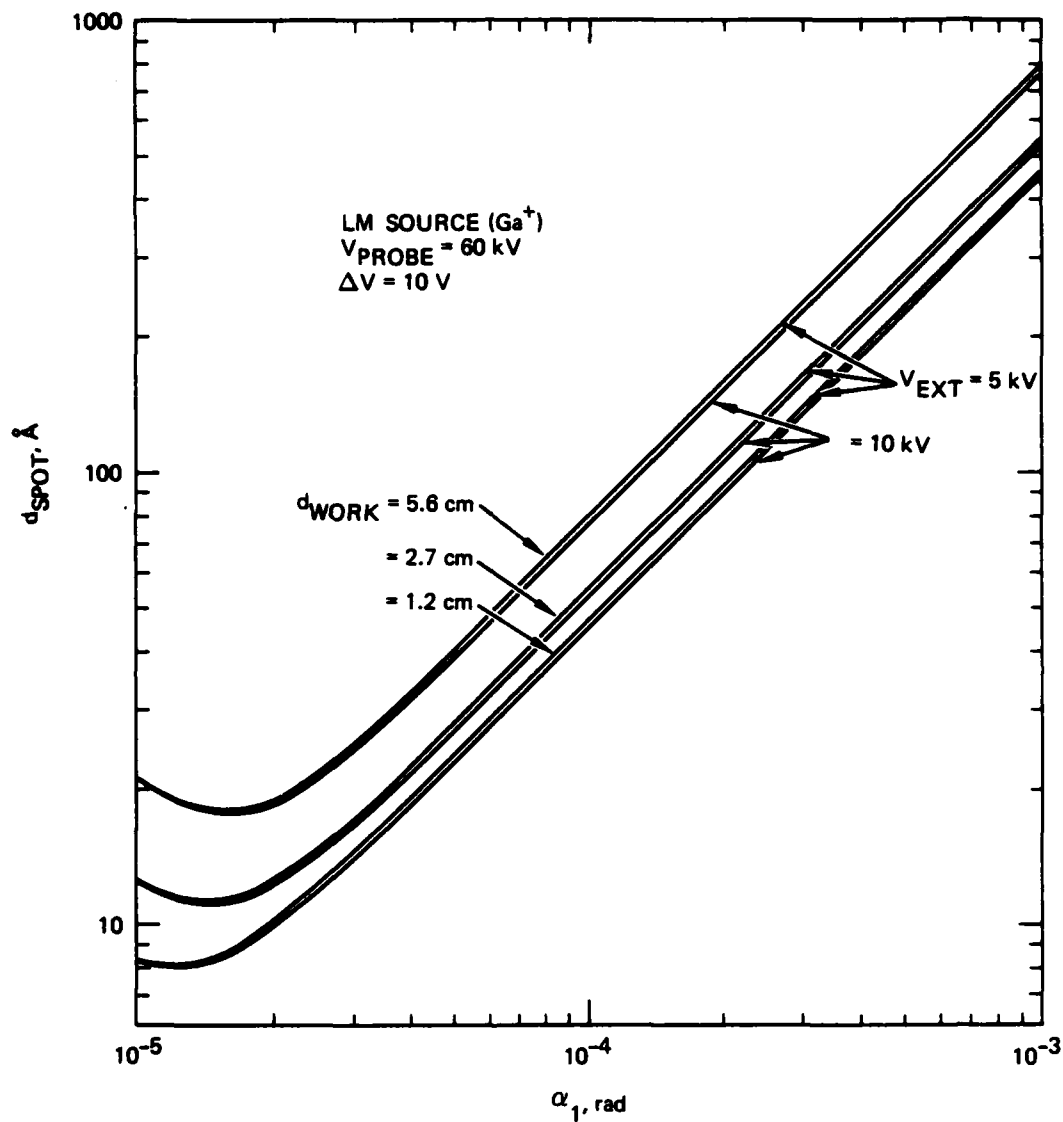


Fig. 14 Spot size as a function of aperture half-angle for LM source omitting effect of virtual source size. At $V_{\text{EXT}} = 5 \text{ kV}$, the overall magnification of the system ranges between ~ 0.1 and ~ 0.4 , increasing with d_{WORK} . At $V_{\text{EXT}} = 30 \text{ kV}$, such range becomes $\sim 0.3 - 0.7$.

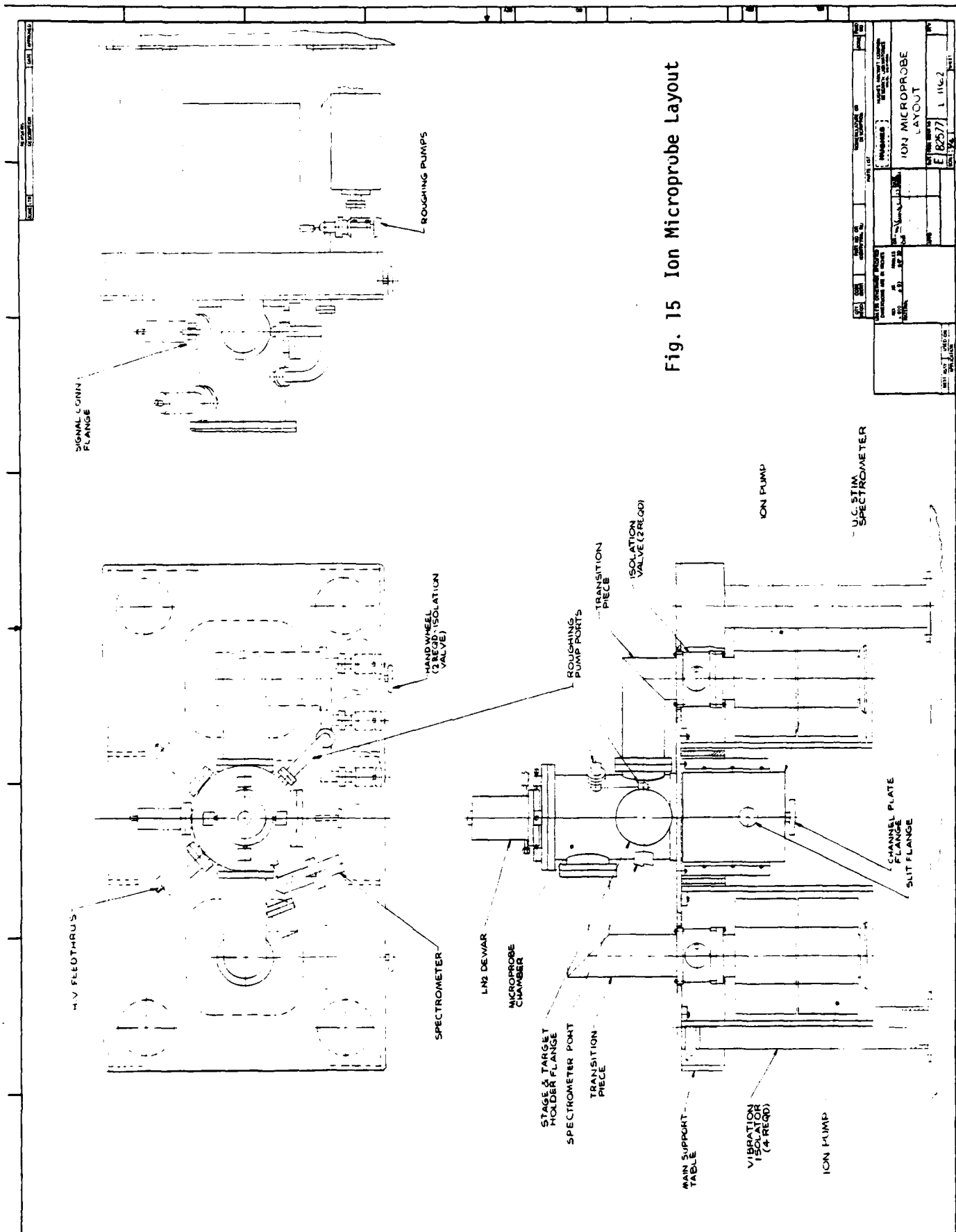


Fig. 15 Ion Microprobe Layout

DATE		BY		CHECKED BY		APPROVED BY	
10/1/78		J. L. HARRIS		J. L. HARRIS		J. L. HARRIS	
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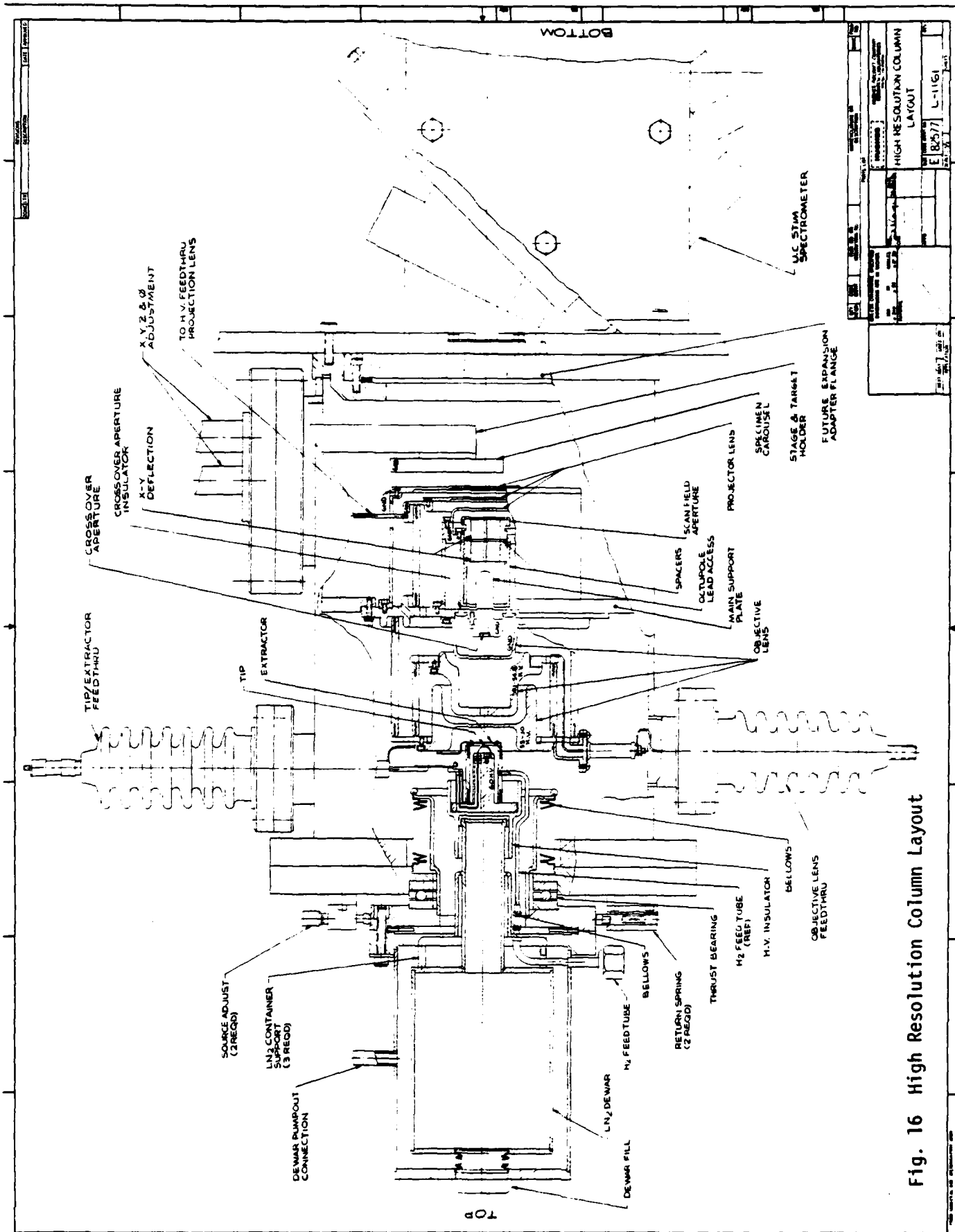
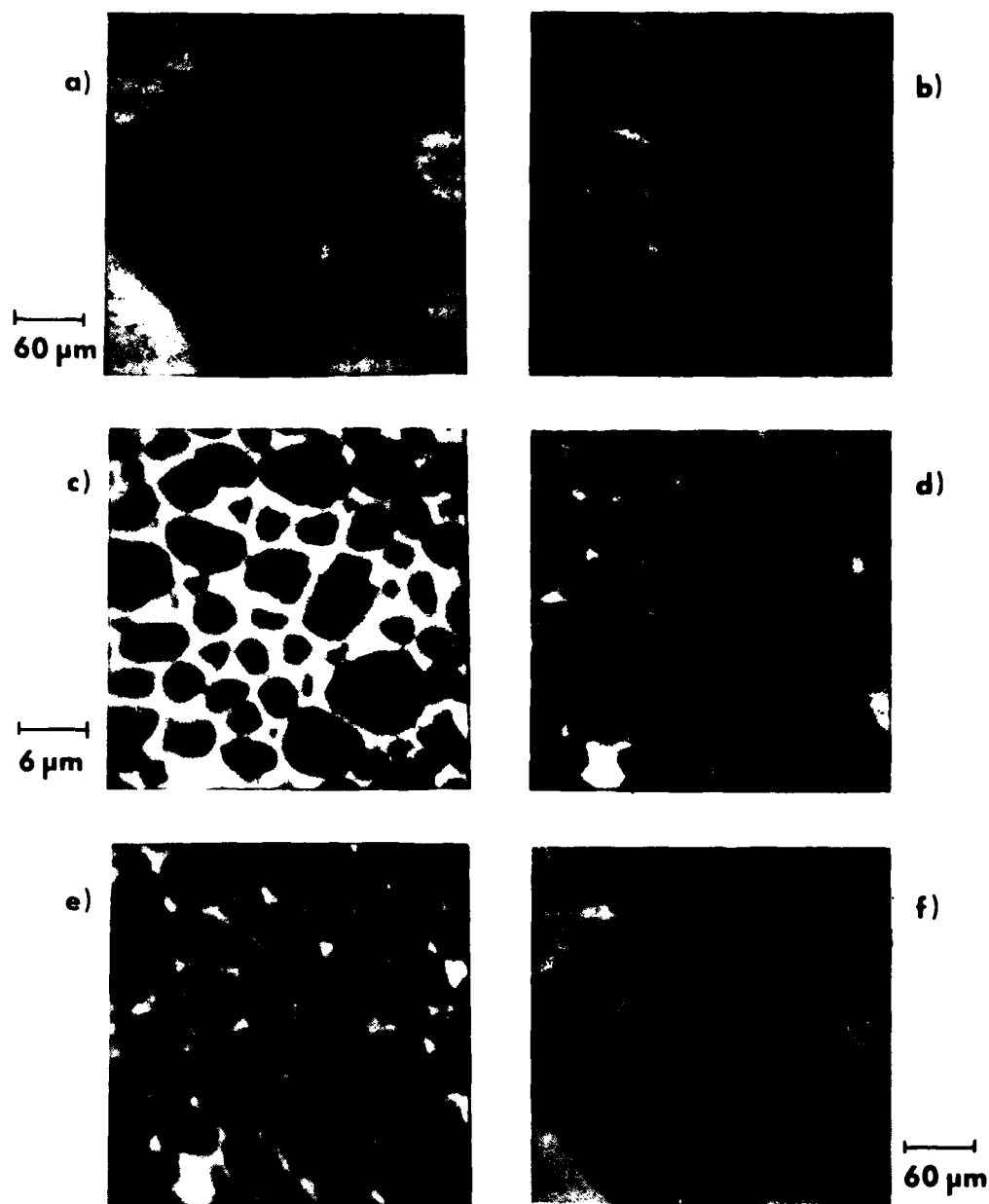


Fig. 16 High Resolution Column Layout



Study of radiation damage (sputtering) induced by 60 keV Ga^+ ion bombardment in the UC-STIM. Micrographs by secondary electron emission during bombardment. Object: 15 Å thick carbon foil supported by holey plastic microgrid. Beam current: $\sim 10^{-11}$ A. Bombardment duration: 90 minutes. Overall dose density: $\sim 4 \times 10^{16}$ ions/cm². Equivalent probe current density for 1 sec: $\sim 7 \times 10^{-3}$ A/cm².

- a) Object before bombardment. Holes in C film appear dark.
- b) through e) Several stages of destruction during bombardment. First the C film is destroyed, then the plastic microgrid is etched until complete destruction of several links.
- f) Damaged region after bombardment.

Fig. 17

